IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of	)	Attorney Docket No.: **MIYOSH0004**
	)	
Tooru SUZUKI et al.	)	Confirmation No.: 3253
	)	
Serial No.: 10/595,134	)	Group Art Unit: 1625
	)	
Filed: March 2, 2006	)	Examiner: Taylor V. OH
	)	
For: METHOD OF SEPARATING STEREOISOMERS OF DICARBOXYLIC ACID HAVING NORBORNENE OR NORBORNANE STRUCTURE, OR DERIVATIVE THEREOF	)	

RULE 1.132 DECLARATION

MAIL STOP: AMENDMENT
U.S. Patent and Trademark Office
Customer Service Window
Randolph Building
401 Dulany Street
Alexandria, VA 22314

Sir:

1. I, Tetsuo YAMANAKA, state that I am over eighteen years old, that I am one of the co-inventors of the subject matter of the above-captioned patent application, and that I am a chemical engineer working for the Quality Assurance Department of Hitachi Chemical Co., Ltd, which is the assignee regarding the above-captioned application. Therefore, I believe that I am an expert in the field of the above-captioned application.

2. I am familiar with the specification and claims of the above-captioned application, including Amendment (C) filed February 18, 2009, and including Amendment (D) filed concurrently with this Declaration. A copy of the claims as amended by Amendment (D) are attached herewith as an Appendix.

3. I have also reviewed the disclosure of U.S. Patent 6,465,551 B1 issued to Zhao et al., of record (hereafter, the "Zhao Patent'). In my opinion, these materials are materials an expert in the field would reasonably consider when forming an opinion regarding the scope of the subject matter disclosed by the Zhao Patent and the scope of the subject matter claimed by the above-captioned application, as amended by Amendment (D).

4. By this declaration, I submit expert testimony and experimental data to show that (1) the Zhao Patent does not disclose a mixture of endo isomer and exo isomer, (2) that even if the Zhao Patent were construed to disclose a mixture of endo isomer and exo isomer, the Zhao Patent does not teach, or suggest, separating the endo isomer and the exo isomer, and (3) that because the Zhao Patent does not teach, or suggest, (1) and (2) above, the Zhao Patent cannot anticipate the subject matter of claims 1-19 of the above-captioned application.

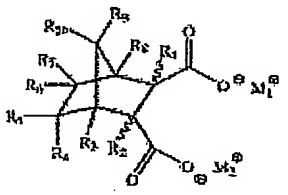
The Claimed Invention

5. The invention of the above-captioned application pertains to a method of separating an endo isomer and an exo isomer of a dicarboxylic acid, or anhydride thereof, wherein the endo isomer is separated from the exo isomer using an alkali metal hydroxide in accordance with the embodiment recited by independent claim 1. Thus, the embodiment according to claim 1 utilizes the difference in solubility between a neutralized salt of the endo isomer and a neutralized salt of the exo isomer of a dicarboxylic acid, which have a norbornene or norbornane structure. This feature of the embodiment according to claim 1 is, in my opinion, not disclosed by the Zhao Patent.

6. With respect to the embodiment of claim 10, a method of separating an endo isomer and an exo isomer of a salt of a dicarboxylic acid is provided that includes steps recited by independent claim 10. Thus, the embodiment according to claim 10 also utilizes the difference in solubility between a salt of the endo isomer and a salt of the exo isomer of a dicarboxylic acid, which have a norbornene or norbornane structure. This feature of the embodiment according to claim 10 is, in my opinion, not disclosed by the Zhao Patent.

The Zhao Patent

7. I have read and studied the disclosure of the Zhao Patent. In my opinion, the Zhao Patent pertains to bicyclo[2.2.1]heptane dicarboxylate salts employed as polyolefin nucleators, which pertain to compounds and compositions comprising specific metal salts of bicyclo[2.2.1]heptane dicarboxylate salts (See Abstract of the Zhao Patent). In particular, the Zhao Patent discloses saturated metal or organic salts of bicyclic dicarboxylates, preferably, bicyclo[2.2.1]heptane-dicarboxylates, and most preferably of a compound conforming to Formula (I):



(I)

wherein M_1 and M_2 are the same or different and are independently selected from the group consisting of metal or organic cations, and R_1, R_2, R_3, R_4, R_5, R_6, R_7, R_8, R_9, and R_{10} are individually selected from the group consisting of hydrogen, C_1 –C_9 alkyl, hydroxy, C_1 –C_9 alkoxy, C_1 –C_9 alkyleneoxy, amine, and C_1 –C_9 alkylamine, halogen, phenyl, alkylphenyl, and geminal or vicinal C_1 –C_9 carbocyclic (Zhao Patent, col. 4, lines 33-56). The Zhao Patent

discloses that, preferably, the metal cations are selected from the group consisting of calcium, strontium, barium, magnesium, aluminum, silver, sodium, lithium, rubidium, and potassium (Zhao Patent, col. 4, lines 56-59).

8. . In Example 3, the Zhao Patent disclosed adding sodium hydroxide (i.e., an alkali metal hydroxide) to a suspension of an endo isomer, namely, endo-bicyclo[2.2.1]hept-5-ene-2,3-dicarboxylic anhydride (Zhao Patent, col. 7, lines 56-67). However, as would be immediately understood by a person of ordinary skill in the art, this suspension contains only the endo isomer and is not a mixture of endo isomer and exo isomer. Thus, Zhao's Example 3 does not teach, or suggest, a mixture of endo isomer and exo isomer, such as is recited by step (a) of claims 1 and 10 of the above-captioned application. In fact, the Zhao Patent discloses that when sodium hydroxide is added to the endo isomer, the result is "[a] clear, homogenous solution...Water was removed in vacuum..." (Zhao Patent, col. 7, lines 61-67). This passage of the Zhao Patent shows, in my opinion, that the endo isomer is dissolved with sodium hydroxide, and then water is removed to obtain a crystalline product. However, the Zhao Patent does not teach, or suggest, in Example 3 separation of an endo isomer and an exo isomer. Therefore, I conclude that Example 3 of the Zhao Patent cannot anticipate the subject matter of claims 1 and 10 because it does not disclose a mixture of endo isomer and exo isomer, and it does not teach, or suggest, separating the endo isomer from the exo isomer as claimed.

9. In Example 2 of the Zhao Patent, Zhao discloses mixing calcium chloride dihydrate to a solution of disodium bicyclo[2.2.1]heptane-2,3-dicarboxylate (Zhao Patent, col.

7, lines 41-55). However, the Zhao Patent does not teach, or suggest, adding alkali metal hydroxide to a solution of disodium bicyclo[2.2.1]heptane-2,3-dicarboxylate. Furthermore, the Zhao Patent does not disclose that the solution employed in Example 2 is a mixture of an endo isomer and an exo isomer. In addition, even if the mixture used in Example 2 were a mixture of endo isomer and an exo isomer, I believe that the Zhao Patent does not disclose the separation of endo isomer from exo isomer.

Experimental Evidence Showing that the Zhao Patent Does Not Disclose Separation of Endo Isomer from Exo Isomer

10. The present experiment demonstrates that the addition of calcium chloride solution to exo and endo isomer of norbornane-2,3-dicarboxylic acid cannot be used to separate the exo and endo isomer if they are together in solution.

Example I

11. Specifically, to 4.72 g of exo-norbornane-2,3-dicarboxylic acid anhydride (0.02 mol), 30 g of water and 20 g of 0.1 mol calcium chloride solution were added together. The mixture was then stirred at 60°C for 2 hours. At that time, exo-norbornane-2,3-dicarboxylic acid anhydride was not dissolved but was present in the suspended form. The suspension was filtered to separate the solid, and the solid was dried. The obtained solid (powder) weighed 4.72 g.

Example II

12. To 4.72 g of endo-norbornane-2,3-dicarboxylic acid anhydride (0.02 mol), 30 g of water and 20 g of 0.1 mol calcium chloride solution were added. The mixture was stirred at 60°C for 2 hours. At that time, endo-norbornane-2,3-dicarboxylic acid anhydride

was not dissolved but was in the suspended form. The suspension was filtered to separate the solid, and the solid was dried. The obtained solid (somewhat agglomerated) weighed 4.68 g.

Discussion of Results

13. As evident from Example I and Example II above, when the endo isomer was in the suspended form by the addition of calcium chloride, it did not dissolve in water. Likewise, when the exo isomer was in the suspended form by the addition of calcium chloride, it also did not dissolve in water. Based on these facts, I conclude that the endo and exo isomers were adsorbed with calcium chloride, respectively. Therefore, the separation of the exo isomer and the endo isomer would be impossible if the endo isomer and exo isomer were both placed together in solution with calcium chloride because, as would be immediately apparent to a person of ordinary skill in the art, it is necessary for one of the endo and exo isomers to be present in an aqueous phase while the other one of the endo and exo isomers is present in a solid phase in order to separate them, one from another, by filtering (Compare, e.g., page 27, lines 18-27, of the specification of the above-captioned application as originally filed).

14. In this case, because the addition of calcium chloride to each of the endo and exo isomers, individually, resulted in a solid phase, it would not be possible to separate the endo isomer from the exo isomer by adding calcium chloride to a solution containing both the endo isomer and exo isomer mixed together, and then filtering the solution, because both the endo isomer and the exo isomer would remain mixed together in the solid phase.

15. By employing exo-norbornane-2,3-dicarboxylic acid anhydride (Example I) and endo-norbornane-2,3-dicarboxylic acid anhydride (Example II) in the above experiment, the experiment is closer to the subject matter of the invention recited by claims 1 and 10 of

the above-captioned application than the subject matter disclosed by the closes prior art, namely, the Zhao Patent.

Conclusion

16. It is my opinion, based on the materials and evidence I have considered, that:

a. the Zhao Patent discloses bicyclo[2.2.1]heptane dicarboxylate salts employed as polyolefin nucleators, and does not teach, or suggest, mixtures of endo and exo isomers;

b. even assuming the Zhao Patent could be construed to teach, or suggest, a mixture of endo and exo isomer (which is, in my opinion, an invalid assumption), the Zhao Patent does not teach, or suggest, separating endo isomer and exo isomer based on mixing them with a solvent (claims 1 and 10), and optionally with an alkali metal hydroxide (claim 1), and then "filtering the mixture...to separate an aqueous phase and a solid phase, thereby separating the endo isomer from the exo isomer" as recited by claims 1 and 10;

c. the addition of calcium chloride to each of the endo and exo isomers of norbornane-2,3-dicarboxylic acid, individually, resulted in a solid phase, so it would not be possible to separate the endo isomer from the exo isomer by adding calcium chloride to a solution containing both the endo isomer and exo isomer mixed together, and then separating the solid phase from the liquid phase; and

d. because the Zhao Patent does not disclose, or even suggest, separating endo isomer and exo isomer based on mixing them with a solvent (claims 1 and 10), and optionally with an alkali metal hydroxide (claim 1), and then "filtering the mixture...to separate an aqueous phase and a solid phase, thereby separating the endo isomer from the exo isomer" as recited by claims 1 and 10, then the Zhao Patent cannot anticipate the subject matter of independent claims 1 and 10.

17. I declare under penalty of perjury that the foregoing is true and correct, that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements so made are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Signed by,

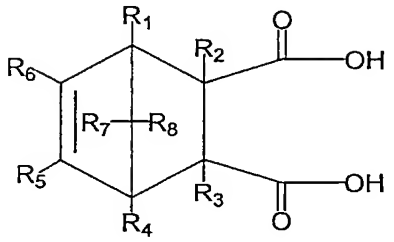
Date: September 1, 2009

Tetsuo Yamanaka

Tetsuo YAMANAKA

APPENDIX:

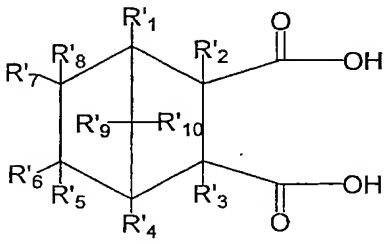
1. A method of separating an endo isomer and an exo isomer of a dicarboxylic acid



(1)

represented by formula (1),

wherein, R_1 to R_8 represent a hydrogen atom, methyl group, ethyl group, or butyl group, or an anhydride thereof, or formula (2),



(2)

wherein, R'_1 to R'_{10} represent a hydrogen atom, methyl group, ethyl group, or butyl group, or an anhydride thereof, the method comprising the steps of:

(a) providing a mixture comprising mainly the endo isomer of the dicarboxylic acid represented by formula (1) or (2) or an anhydride thereof, and the exo isomer of the dicarboxylic acid represented by formula (1) or (2) or an anhydride thereof;

(b) mixing the mixture with an alkali metal hydroxide and a solvent; and

(c) filtering the mixture obtained in step (b) to separate an aqueous phase and a solid phase, thereby separating the endo isomer from the exo isomer.

2. The method of separating an endo isomer and an exo isomer according to claim 1, wherein said dicarboxylic acid consists essentially of a dicarboxylic acid represented by the formula (1) or an anhydride thereof.

3. The method of separating an endo isomer and an exo isomer according to claim 2, wherein the alkali metal hydroxide is used in a quantity that achieves at least 0.2 equivalents relative to the endo isomer, and no more than 8 equivalents relative to the mixture.

4. The method of separating an endo isomer and an exo isomer according to claim 2, wherein the solvent is used in a quantity of at least 0.7 g relative to 6 mmol of the mixture, and no more than the larger of either 10 g relative to 6 mmol of the mixture or 20 g relative to 6 mmol of the endo isomer.

5. The method of separating an endo isomer and an exo isomer according to claim 2, wherein the dicarboxylic acid represented by formula (1) or an anhydride thereof is 5-norbornene-2,3-dicarboxylic acid or an anhydride thereof.

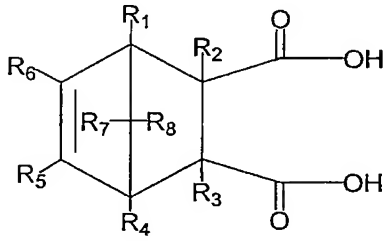
6. The method of separating an endo isomer and an exo isomer according to claim 1, wherein said dicarboxylic acid consists essentially of a carboxylic acid represented by formula (2) or an anhydride thereof.

7. The method of separating an endo isomer and an exo isomer according to claim 6, wherein the alkali metal hydroxide is used in a quantity that achieves at least 0.35 equivalents and no more than 8 equivalents relative to the mixture.

8. The method of separating an endo isomer and an exo isomer according to either claim 6, wherein the solvent is used in a quantity of at least 0.7 g relative to 6 mmol of the mixture, and no more than 20 g relative to 6 mmol of the mixture.

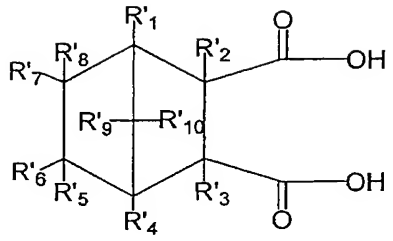
9. The method of separating an endo isomer and an exo isomer according to claim 6, wherein the dicarboxylic acid represented by formula (2) or an anhydride thereof is norbornane-2,3-dicarboxylic acid or an anhydride thereof.

10. A method of separating an endo isomer and an exo isomer of a salt of a dicarboxylic acid represented by formula (1),



(1)

wherein, R_1 to R_8 represent a hydrogen atom, methyl group, ethyl group, or butyl group, or formula (2),



(2)

,

wherein, R'_1 to R'_{10} represent a hydrogen atom, methyl group, ethyl group, or butyl group, the method comprising the steps of:

(a) providing a mixture comprising mainly the endo isomer of the salt of the dicarboxylic acid represented by formula (1) or (2), and the exo isomer of the salt of the dicarboxylic acid represented by formula (1) or (2);

(b) mixing the mixture with a solvent; and

(c) filtering the mixture obtained in step (b) to separate an aqueous phase and a solid phase, thereby separating the endo isomer from the exo isomer.

11. The method of separating an endo isomer and an exo isomer according to claim 10, wherein said salt of a dicarboxylic acid consists essentially of a salt of a dicarboxylic acid represented by formula (1).

12. The method of separating an endo isomer and an exo isomer according to claim 11, wherein the salt of the dicarboxylic acid represented by formula (1) is a salt of 5-norbornene-2,3-dicarboxylic acid.

13. The method of separating an endo isomer and an exo isomer according to claim 10, wherein said salt of a dicarboxylic acid consists essentially of a salt of a dicarboxylic acid represented by formula (2), with a solvent.

14. The method of separating an endo isomer and an exo isomer according to claim 13, wherein the salt of the dicarboxylic acid represented by formula (2) is a salt of norbornane-2,3-dicarboxylic acid.

15. The method of separating an endo isomer and an exo isomer according to claim 1, wherein step (c) is a step of filtering a mixture obtained from the mixing step, and either obtaining an endo isomer of a salt of the dicarboxylic acid represented by formula (1) or (2) as a liquid phase, or obtaining an exo isomer of a salt of the dicarboxylic acid represented by formula (1) or (2) as a solid phase.

16. The method of separating an endo isomer and an exo isomer according to claim 15, further comprising the step of obtaining an endo isomer or an exo isomer of the dicarboxylic acid represented by formula (1) or (2), from the endo isomer or the exo isomer of the salt of the dicarboxylic acid represented by the formula (1) or (2).

17. The method of separating an endo isomer and an exo isomer according to claim 15, further comprising the step of obtaining an endo isomer or an exo isomer of an anhydride of the dicarboxylic acid represented by formula (1) or (2) from the endo isomer or the exo isomer of the dicarboxylic acid represented by formula (1) or (2) or a salt thereof.

18. An endo isomer of a dicarboxylic acid represented by formula (1) or (2) or a derivative thereof, obtained using the method according to claim 1.

19. An exo isomer of a dicarboxylic acid represented by formula (1) or (2) or a derivative thereof, obtained using the method according to claim 1.